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ELECTRODEPOSITION PAINTING OF METAL SUBSTRATES, (U)  
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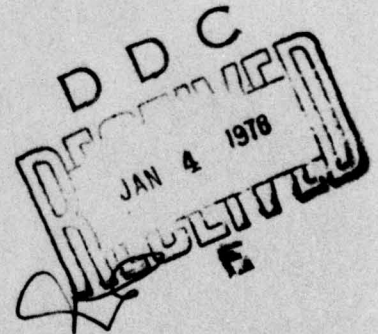
**REPORT**

**MRL-R-693** ✓

**ELECTRODEPOSITION PAINTING OF METAL SUBSTRATES**

**J.A. Gagliardi and D.J. Whelan**

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10 J.A./Gagliardi D.J./Whelan

ABSTRACT

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Various water-borne electrodeposition paints based on the acrylic resins, Baycrl L461W and Cyanamid Resin XC-4010, were formulated and properties of the films deposited onto various metal substrates from these paints and from an Australian-made paint, Dulux Light Grey Electrocoat, have been studied in detail. Durable films with good adhesion, scratch and impact resistance are deposited on mild steel, on aluminium and on painted panels, although the surface finish on both aluminium and tin-coated steel is less smooth than that on steel.

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Various water-borne electrodeposition paints based on the acrylic resins, Baycrl L461W and Cyanamid Resin XC-4010, were formulated and properties of the films deposited onto various metal substrates from these paints and from an Australian-made paint, Dulux Light Grey Electrocoat, have been studied in detail. Durable films with good adhesion, scratch and impact resistance are deposited on mild steel, on aluminium and on tin-plated panels, although the surface finish on both aluminium and tin-plate is less smooth than that on steel.

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## **ELECTRODEPOSITION PAINTING**

### **OF METAL SUBSTRATES**

#### **1. INTRODUCTION**

Interest at these laboratories in the electrodeposition of organic coatings was prompted by a request for advice on a preferred method of coating steel cylinders, 35 mm x 110 mm in size, closed at one end. The requirements specified in this task prompted a review of various methods of paint application, that of electrodeposition appearing the most suitable.

##### **1.1 Requirements**

The following requirements were considered desirable :

- (i) the storage life of the coating under shelter, over a wide range of temperature and humidity, was to be twenty years,
- (ii) the dried coatings were not to affect metals or organic materials stored either in contact with the coated cylinders or nearby them,
- (iii) the coatings were to be resistant to impact and abrasion,
- (iv) the coated surface was to be smooth and blemish free, and
- (v) the coating thickness was to be substantially uniform over the whole interior and exterior surfaces and to be between 20 and 25 microns.

##### **1.2 Comparison of Methods of Application (1)**

The following techniques were considered with respect to the above requirements.

###### **(i) Conventional Air Spraying**

Previous experience indicated that it would be difficult to obtain the required film thickness tolerances and evenness of coating, particularly on the inner surfaces, by this method.



### *(ii) Dipping*

Because of the "run off" associated with this method, an uneven coating always resulted and tears often occurred.

### *(iii) Wet Electrostatic Spraying*

Trials showed that this was a possible method, particularly if the cylinders were spun during application. However, the "throwing power" of the paints normally available is not sufficient to coat the whole of the interior of the cylinders satisfactorily.

### *(iv) Powder Coating*

Trials showed that the film thicknesses obtained by powder coating were generally in excess of those requested and that it was difficult to meet the required film thickness specification.

### *(v) Electrodeposition (also known as Electrophoretic Coating)*

According to the literature, this appeared to be a suitable method as both the interior and the exterior could be coated simultaneously, apparently with good control over the film thickness.

In the original request, it was stipulated that coatings on the cylinders must not produce corrosive vapours on prolonged storage. This precluded the use of oil-based paints as previous work had shown that coatings from these paints can emit undesirable by-products on storage (2). Hence, of the electrodeposition paints and resins available, it appeared that the requirements would be best served by an acrylic-based formulation.

Accordingly, the deposition onto mild steel and other substrates of paints prepared from two commercially-available acrylic-based resins, Baycrl L461W (Bayer) and Cyanamid Resin XC-4010 (American Cyanamid), was investigated. The properties of films from these paints were compared with those from a locally manufactured product, Dulux 650-11983 Light Grey Electrocoat, a pigmented epoxyester formulation.

## 1.3 Basis of Electrodeposition (3)

There are two types of electrocoating technique, anodic electrodeposition and cathodic electrodeposition. Of these, anodic electrodeposition (4) was developed first on a commercial scale, although, more recently, a few industrial plants based on cathodic electrodeposition have been installed for certain special applications (5).

In simple terms, the technique is a paint-plating operation where the object to be coated, which must be conductive itself, is dipped into a tank of water-thinned paint and an electric current passed through the system. In anodic electrodeposition, most of the paint vehicles used are carboxylated versions of conventional thermosetting resins. These resins are either solubilised or dispersed in water by the addition of bases, such as amines; the negatively-charged paint solids migrate (hence the name electrophoretic coating) to the object to be coated which forms the anode.

Ideally, the paint "plates out" over the surface of the anode, leaving a uniform film of firmly bound paint of relatively low water content adhering to it. The object is then thoroughly rinsed with clean water, air-dried and cured in a conventional stoving oven.

Present plants which operate anodic electrodeposition facilities use a bath with a final total solids content of about 10 per cent by weight and typically achieve final film thicknesses of approx. 25  $\mu$ m (1 mil), at electrodeposition voltages of 100 volts applied for one minute.

The major difference between electroplating of metal and electrocoating of paint films is that the deposited organic film acts as an electrical insulator over the surface covered; subsequent further deposition then occurs preferentially on the more conducting "exposed" metal surface. As the paint film builds up on the anode, the current (under constant applied voltage) diminishes to a very low value.

Under these conditions, electrodeposition can be directly used to apply only a thin single coating, be it primer or a decorative, functional finish, onto a conducting surface. In many instances, thicker films may be desired and, to this end, novel formulations are being investigated overseas which may enable the technique to be adapted to produce both thicker coatings and two-coat systems (6,7).

Electrodeposition appears to offer many advantages. The paints used are based on water-borne coating formulations (8) and the technique gives smooth, uniform films over the whole surface, including edges, corners and crevices. On the other hand, electrodeposition facilities are expensive to set up, and, for successful operation, technical skill and judgement are needed to maintain the coating bath.

## 2. EXPERIMENTAL

### 2.1 Resins

Among the acrylic resins available locally, two were chosen which were suitable for electrodeposition purposes, Baycrl L461W (Bayer) (9) and Cyanamid Resin XC-4010 (American Cyanamid) (10).

Previous investigations (11) and trade literature (9,10) indicated:

(1) Baycrl L461W is a self reacting thermosetting acrylic resin, 55 per cent solids in isobutanol, manufactured by Bayer AG, Leverkusen, West Germany. It has an acid value of 65-75.

From the application of UV, IR and  $H^1$  - nmr spectroscopy and from pyrolysis - gas chromatography - mass spectroscopy, it has been established that the resin is a copolymer, containing styrene, acrylic acid and an acrylic ester (probably butyl acrylate). Nitrogen is also present (Lassaigne test), probably as an amide (IR spectrum) (11).



As large quantities of this resin were available, most of the development work was carried out using this resin.

(11) In order to develop satisfactory film properties from Cyanamid Resin XC-4010, it is recommended that the resin be codeposited on the substrate with an amino cross-linking agent, such as Cymel XM-1116 (10(d)). Both of these are produced by American Cyanamid Co., Stamford, Connecticut, U.S.A.

Cyanamid Resin XC-4010 has been described by the manufacturer as an aliphatic anionic resin, 73-76% solids in ethylene glycol monomethyl ether, and with an acid value 95-115. Its infra-red absorption spectrum has broad absorption bands (liquid film) at  $1740\text{ cm}^{-1}$  (carboxylic ester) and  $1705\text{ cm}^{-1}$  (carboxylic acid); this is consistent with published material (10(c)) which suggests that the resin is a copolymer of an acrylate and acrylic acid. The infra-red spectrum also contains absorption bands characteristic of pendant benzenoid groups at  $1600\text{ cm}^{-1}$ ,  $1585\text{ cm}^{-1}$ ,  $1500\text{ cm}^{-1}$  and at  $755\text{ cm}^{-1}$  and  $710\text{ cm}^{-1}$ ; this suggests the presence of polymerized styrene groups in the polymer and is confirmed by the appearance of benzenoid absorptions in the UV spectrum ( $\text{CH}_3\text{OH}$  as solvent) superimposed on a broader, more intense band which extends beyond  $230\text{ nm}$ . The presence of styrene has been confirmed by pyrolysis - gas chromatography - mass spectroscopy (11(a)).

The relative intensities of the benzenoid absorptions in the UV spectra suggested that the concentration of aromatic groups in Baycrl L461W, was about twice that in the Cyanamid products.

The cross-linking agent, Cymel XM-1116, is supplied as a colourless viscous liquid and is reported to be a full alkylated hexamethylolmelamine. The  $^1\text{H}$  nmr spectrum of this product ( $\text{CDCl}_3$  as solvent) has resonances (relative to TMS) at  $\delta$  (ppm) 1.15 (triplet,  $J = 7\text{ Hz}$ ,  $3\text{H}$ ,  $\text{CH}_3 - \text{CH}_2 - \text{X}$ ), 3.30 (singlet,  $2\text{H}$ ,  $\text{X}-\text{CH}_2-\text{Y}$ ) and 3.52 (quartet,  $J = 7\text{ Hz}$ ,  $2\text{H}$ ,  $\text{CH}_3 - \text{CH}_2 - \text{X}$ ); this indicates that the alkoxyl group is, in fact, the ethoxyl group, the integration of the spectrum corresponding to one ethoxyl group per alkoxy-methyl group, ( $\text{N} - \text{CH}_2 - \text{O}$ ). There are also a broad absorption at  $\delta$  5.74 (ca. 0.03 H) due to residual N-H groups and two unidentified peaks at  $\delta$  5.10 and 5.15 ppm (combined integrated intensity ca. 3H).

(11i) Dulux 650-11983 Light Grey Electrocoat is a pigmented epoxyester formulation.

## 2.2 Formulations

Thermosetting electrodepositions paints are similar in basic composition to conventional paints and consist of the following :

- (a) resin
- (b) pigment
- (c) neutralising agent
- (d) carrier solvent

Paints based on the Baycryn resin were formulated as clear and as pigmented paints.

Several white formulations were made up varying only the amount of rutile titanium dioxide (Tioxide RCR 6, supplied by Tioxide Australia Pty.Ltd.). These paints have been designated "Baycryn White n" where n is the weight of titanium dioxide used in the formulation relative to 160 g Baycryn L461W as supplied. The formulation of Baycryn White 36 illustrates the procedure, (Appendix A).

Preliminary investigations indicated that thicker coatings with improved gloss and smoothness were obtained by the addition of small amounts, between 0.5 and 2 per cent, of higher alcohols (e.g. isodecanol), to the bath. These properties were also enhanced in the deposited film by the use of alkanolamines, for example, NN-dimethylethanolamine, as neutralising (solubilising) base rather than by the use of ammonia, dimethylamine or trimethylamine for this purpose.

The paint designated Baycryn White 36/6 was prepared in a manner similar to that for Baycryn White 36 - the additional character /6 designating the relative volume of isodecanol incorporated in the formulation at the milling stage, (Appendix B).

Cyanamid paints and Dulux Light Grey Electrocoat were prepared according to published recommendations, (Appendix C).

### 2.3 Substrates

Rectangular panels,  $15 \times 6.7 \times 0.1 \text{ cm}^3$ , were cut from mild steel; similar panels were also prepared from aluminium and from tin-plated steel. All panels were sanded to present a bright surface and rubbed clean with a moist cloth to remove the fine dust of metal adhering to the surface, dried, degreased and desmutted by rubbing with mineral turpentine. This procedure conforms to Australian Standard AS 1580 (May 1975), "Pretreatment of Metal Test Panels".

### 2.4 Electrodeposition

A stainless steel panel was used as the cathode, the test substrate forming the anode. The distance between the anode and the cathode was normally 5 cm. Before each run, the contents of the bath, contained in a two litre glass beaker, were agitated vigorously (Torrance Stirrer) to disperse any separated material; there was no adverse settling of material during the course of the electrodeposition, up to five minutes.

All electrodepositions were run at constant voltage. Although the output from the power supply could be altered from 0-100 volts DC, most of the work described here was carried out at 60 volts, for electrodeposition times up to five minutes. At this voltage, satisfactory film thicknesses were built from formulations based on each of the two resins and the commercial paint, while adverse effects on the deposited films (e.g. blistering, cratering, etc.) from reactions at the anode were minimised.



After deposition, the coated panels were removed from the bath and washed with running water to remove loosely adhering excess paint ("drag off") which results solely from contact of the panel with the bath and has not been deposited by electrodeposition. At this juncture, coatings from all three resins were tacky and soft to touch. Those coatings from Cymel-based formulations were easily damaged and deformed by contact while those from Dulux Light Grey Electrocoat and from Baycryn-based formulations were much less plastic and consequently easier to handle. The washed panels were air-dried overnight at ambient temperature and then baked at 140-160°C for thirty minutes.

Compromises had to be made to achieve reasonable hiding power, film thickness and gloss from the deposited films. Lower pigment concentration in deposition bath gave a coating with poor hiding power but higher pigment concentrations resulted in lower gloss coatings. By the same token, when shorter electrodeposition times were employed, thinner films of higher gloss were usually obtained but these films had poorer hiding power.

## 2.5 Physical Measurements

Several physical measurements were made on the deposited films. These were :

### (1) *Film Thickness*

This property was measured using Fischer "Permascope Non-destructive Film Thickness Testers", Type ES (for mild steel panels and tin-plated steel panels) and Type EC Model 7034 (for aluminium panels and copper panels).

### (2) *60° Gloss*

Gloss was measured in accordance with the specification of the Standards Association of Australia, Australian Standard AS 1580, Method 602.2 (April, 1974), using a Gardner Instruments PG-5500 Digital Photometric Unit fitted with a 60° Precision Glossmeter Head.

### (3) *Scratch Resistance*

Scratch resistance was determined following the method conforming to the specification of the Standards Association of Australia, Australian Standard 1580, Method 403.1 (May, 1975), using an A.I.D. Automatic Scratch Test Machine for Paints, (Series No. 6978), manufactured by Sheen Instrument Ltd., Surrey, England.

In principle, the scratching medium is a loaded tungsten carbide spherical point, 1 mm in diameter and the scratch resistance is the maximum load (in g) that the film will withstand scratching through to the substrate; it is a function of both the thickness of the film and the nature of the film.

### (4) *Adhesion*

This property was determined using an Epprecht Twist-o-meter (Epprecht Industries, Switzerland).

Dollies compatible with the instrument were stuck to the coated substrate with Araldite Black/Blue Adhesive (Ciba-Geigy), which was allowed to cure for four days at ambient conditions. Adhesion is measured as the stress intensity necessary to break the surface of the coating from the substrate. It assumes that there will be greater adhesion in the dolly-adhesive-coating than in the coating-substrate system.

The instrument is calibrated in units of  $\text{kg wt cm}^{-2}$ .

#### (5) Impact

This test conformed to that specified by the American Society for Testing and Materials, Standard D 2794-69 (October, 1969), using a Gardner Impact Tester, Model IG 1120.

#### (6) Bend Test

This test followed the method specified by the Standards Association of Australia, Australian Standard 1580, Method 402.1 (May, 1975), using a Sheen Bend Test Apparatus, No. 802 (Sheen Instruments Ltd., Surrey, England), employing 1/8", 1/4" and 1/2" mandrels at 0°C and 25°C.

### 2.6 Durability

Electrocoated mild steel panels were exposed to both salt-spray tests and accelerated weathering tests and the performance of these painted panels was compared with that from spray-painted reference panels, one prepared from a solvent-based acrylic paint, the other from an epoxyster paint.

#### (i) Salt-Spray Test (12)

A cross was scratched across the paint film of each panel to expose bare metal and to simulate a damaged surface. The panels were then exposed to the Salt-Spray Test (DEF-1053, No. 24) (12) for two hundred hours.

At the conclusion of the test, both the panels and the scratch lines were assessed for rusting and for blistering of the paint film. For the panels, the performance of the film was rated on a scale "10-to-0", where a rating of "10" indicates "no failure" and "0" indicates "complete failure"; along the scratch lines, assessment of rust was in terms of "slight", "medium" or "heavy".

#### (ii) Accelerated Weathering

An Atlas Weatherometer (Carbon Arc) was used as the artificial weathering medium. Panels were exposed for a period of 1000 hours under conditions conforming to the ASTM procedures (13).

Chalking and rusting of the paints were assessed on a scale "10-to-0" as before. Colour changes (as AN42 units) were calculated from chromaticity measurements made on the panels before and after exposure, using a differential colorimeter (14).



### 3. RESULTS

The following criteria were adopted to evaluate the performance of the cured paint films :

- (i) film thickness
- (ii) gloss (60 degree) and smoothness
- (iii) appearance, including whiteness and hiding power (subjective)
- (iv) scratch resistance
- (v) adhesion, and
- (vi) durability

#### 3.1 Preliminary Work

Initial experiments were carried out on a simple Baycryn-based formulation, consisting of :

Baycryn L461W (55% in butanol)	491.2 g
Neutralising Amine	17.8 g
Pigment (TiO <sub>2</sub> )	134.0 g
Distilled water	357 ml

The final pH of this formulation was ca. 7.4. Additives were incorporated as required.

These experiments confirmed many facts about the process which are covered in the literature (3,15). These will not be repeated here, save one, the dependence of the condition of the cured, deposited film on the grade of titanium dioxide (16) used in the paint formulation.

The finishes of Baycryn films deposited from paints pigmented with uncoated rutile, Tioxide RSM2, and anatase, Tioxide AE, were rough, badly cratered and pin-holed compared with those pigmented with the more refined and coated grades of rutile, Tioxide RCR2 and RCR6.

It is well known that the presence of inorganic ions in an electro-deposition bath can alter the paint deposition process at the anode, leading to defective films.

Conductivity measurements on aqueous dispersions of the four grades of titanium dioxide used were made and are presented in Table 1. The results show that the conductivity of the more refined RCR2 and RCR6 is less than that from the uncoated RSM2 and AE grades, and confirm that there is a greater concentration of inorganic ions on these latter pigments.

### 3.2 Developmental Work

Mild steel panels were coated under similar conditions from baths of Baycryl-based paints of different formulations, based on Baycryl White n (where n has been previously defined, Section 2.2).

It was found that the coatings from formulations with lower  $\text{TiO}_2$  (Rutile RCR6) contents, for example, Baycryl White 12, (pigment : binder, P:B = 0.075), and Baycryl White 24, (P:B = 0.15), had lower film thicknesses, poorer hiding power and whiteness but higher gloss than those coatings from baths formulated with a higher  $\text{TiO}_2$  (Rutile RCR6) content, for example, Baycryl White 30; adhesion was similar from all Baycryl-based formulations investigated - the best combination of properties being obtained from the formulations Baycryl White 30 (P:B = 0.19) and Baycryl White 36, (P:B = 0.22). Thicker films were obtained as P:B ratio increased so most detailed work was carried out using formulations based on Baycryl White 36. The gloss and adhesion on panels from Baycryl White 30 and Baycryl White 36 were similar and were higher than that from Baycryl White 40.

Subsequent developmental work with Cyanamid-based formulations and from Dulux Light Grey Electrocoat followed these initial observations.

### 3.3 Physical Properties of Deposited Films

Results summarising some of the more important physical properties of the electrodeposited films are presented in detail in Tables 2 to 12. In addition, micrographs of the surfaces of various electrodeposited coatings (magnification x 25) illustrate various pertinent features.

In all cases, the best coatings are deposited from freshly prepared baths. This is shown in Table 13 where the ageing of both Baycryl and Cymel based paints are compared. Not only did the mass of paint deposited decrease but the appearance of the films, as described by gloss, hiding power and coverage, deteriorated.

In general, the various electrodeposition paints perform favourably when compared with solvent-based epoxyester- and acrylic-based paints applied by conventional spray painting, (Tables 14-16). The electrodeposition paints exhibit superior scratch resistance, and flexibility (bend test), good adhesion to mild steel and satisfactory impact resistance; their durability is also comparable to that of the spray-coated finishes, (see Section 3.6).

### 3.4 Electrodeposition Paint Films

#### 3.4.1 Cymel Electrodeposition Paints

Coatings on mild steel panels from Cymel-based formulations are tacky and easily damaged when removed from the electrodeposition bath. However, after stoving at 140-150°C and cooling, the panels present a firm, smooth coating, quite free of blemishes (Fig. 1). The pigmented coating has good hiding power and high gloss and both the pigmented and the clear coatings exhibit good adhesion, scratch resistance and impact resistance.



### 3.4.2 Dulux Light Grey Electrocoat

Uncured coatings from Dulux Light Grey Electrocoat are less tacky and more robust than those from Cymel formulations. On stoving at 140°-160°C, the coating cures to present a matte, well covered, smoothly-finished surface, which is resistant to water, to acid and alkali and to common organic solvents (methylene chloride, acetone and toluene). When viewed under a microscope (x 25), the surface appears densely coated with few irregularities, pinholes, craters or blemishes, (Fig. 2).

### 3.4.3 Baycrl Electrodeposition Paints

Similar satisfactory surface coverage also develops on mild steel panels covered with Baycrl Clear and with Baycrl Red, (Fig. 3), although upon stoving at 140-160°C, the colour of Baycrl Red panels darkens; this is probably due to slight thermal breakdown of the pigment - Monolite Fast Red GS (ICI), an azo dye from the coupling of the diazonium salt of 2,4-dinitroaniline with 2-naphthol.

Panels coated from baths prepared with Baycrl White formulations show uniform coverage with good hiding power and good gloss, but on mild steel, on aluminium and on tin-plated panels, they exhibit a perceptible roughness. This roughness is akin to the "dried spray effect" observed on the surface of spray-painted substrates when evaporation of solvent has occurred too rapidly. It is most obvious in highly pigmented coatings.

Examination of the surfaces of unstoved and stoved coatings from Baycrl White 36 deposited on mild steel panels is informative. The surface of the unstoved coatings is very similar to that of the stoved coatings and confirms the observation that there is negligible flow in Baycrl White panels on stoving at 140°-160°C. This contrasts with coatings from Cymel White which do flow on stoving.

Micrographs of the surfaces of stoved Baycrl panels are presented in Figs. 4, 5 and 6. Initially, the paint film builds up rapidly at an applied voltage of 60 V to deposit a thin, pigmented film over the whole surface. After an electrodeposition time of one minute, the surface of the coating, after stoving at 140-150°C, is intact but exhibits microscopic cratering. However, the coating is quite durable and protects the coated substrate from attack by galvanic action from copper sulphate solution.

As electrodeposition proceeds further, the thickness of the deposited films from Baycrl White 30 and Baycrl White 36 increases but very small pinholes appear on the surface of the coatings, (Fig. 6). They are still present after stoving. Microscopically observable cratering is also more pronounced.

The pinholing observed in the Baycrl finishes probably originates from two sources (17) :

- (1) rupture of the deposited film by oxygen liberated at the anode during the electrolytic reaction which accompanies the electrocoating process, and

- (11) from the physical process of film deposition, which is evidently an exothermic reaction (17).

Fortunately, recoating takes place at these points where film damage has occurred so the durability of the Baycryn-finished surface and the good performance noted under conditions of the salt-spray test (Table 15) is not seriously impaired.

Cratering originates from a selective separation of the components of a deposited coating as the composition of the medium alters, often by solvent evaporation (18). In the present instance, it probably arises from the rapid and efficient dehydration of the deposited film by electroosmosis (19).

Both pinholing and cratering are much less pronounced in the reformulated preparation, Baycryn 36/6, (Fig. 7), a preparation in which a greater quantity of isodecanol is dispersed throughout the medium by in-situ grinding at the manufacturing stage. Coatings from Baycryn 36/6 are more tacky than those from Baycryn 36 prior to curing but, after stoving, they present a more glossy, homogeneous surface, (Fig. 7). Their physical properties indicate that thicker films are thrown from Baycryn 36/6 but both adhesion and scratch resistance are somewhat decreased.

The coatings thrown from Baycryn 36/6 onto aluminium and onto tin-plated panels (Fig. 8 and 9) show similar properties to those thrown onto steel panels; in fact, the "dried spray effect" is even more pronounced on aluminium surfaces, (Fig. 8).

### 3.5 Bath Stability

As mentioned earlier, the quality of the coating deposited does depend on the age of the electrodeposition paint. The Baycryn resin and the Cyanamid resin are both copolymers containing ester and carboxylate groups; in an aqueous medium at pH 8 and in the presence of nucleophilic reagents, hydrolysis of the ester groups will take place (20) and this may reflect itself in the deposition of coatings from aged baths with properties different from those of freshly-prepared baths. Indeed, this occurs and it has been observed that the coatings thrown onto mild steel panels from well-dispersed aged baths from Baycryn White 36 and Baycryn White 36/6 are more coarse and more uneven than those from freshly prepared baths. This is illustrated by a comparison of Figs. 10 and 11 with Figs. 6 and 7. In addition, bath stability has been assessed in terms of the variation in the mass deposited per unit area of panel covered with the age of the bath, (Table 13). After twenty-eight days, the mass deposited per unit area on mild steel panels from Baycryn White 36/6 was 60% of that deposited originally from a freshly prepared bath, while that from Cymel White was 75%. The baths had been stored at 19-22°C over this period and were used solely to test bath stability in terms of this ageing effect.

By the same token, the durability of the deposited films from these aged baths is not seriously impaired compared with those of similar film thickness deposited from freshly prepared baths.



### 3.6 Durability of Electrodeposition Films

In Tables 15 and 16, the performance of electrocoated panels subjected to salt spray testing and artificial weathering is assessed and the results compared with those obtained from spray-painted epoxyester- and acrylic-based formulations.

In the Salt Spray Test, the thicker coatings from a given formulation afforded greater protection to the mild steel substrates than did the thinner coatings. Good protection was obtained from both the Cymel White and the Baycrl White formulations, their performance surpassing that of the spray-painted coatings. The least satisfactory paint was the Dulux Light Grey Electrocoat, where much thinner coatings were deposited at 60 volts, (Table 12). This may be related to an ion-exchange or leaching reaction between the pigment (an inorganic chromate) and the salt spray medium ( $\text{NaCl}$ ,  $\text{MgCl}_2$ ,  $\text{CaCl}_2$ , etc.), rather than to any inherent susceptibility of the resin, as the Dulux coating displays very good resistance to solvents, acid (1M  $\text{HCl}$ ) and alkali (0.5M  $\text{NaOH}$ ).

The solvent-based spray-painted epoxyester- and acrylic-based paints show excellent resistance to artificial weathering, being superior to the Baycrl- and Cymel-based electrodeposition paints. The Baycrl paint displays good resistance to chalking and to rusting but considerable loss of gloss occurs. Overall, the Dulux Light Grey Electrocoat also performs well in this test. The Cymel paint is least satisfactory, having poor chalking resistance accompanied by considerable "loss of gloss".

In meeting the requirements of the original request, resistance to weathering was not a stringent demand but one which should be borne in mind should the circumstances demand it.

## 4. DISCUSSION

Many factors effect the quality of the film deposited on a metal surface; in the present instance, the electrodeposition of several coating formulations on mild steel panels has been studied in detail with the requirements of the original request in mind. The material presented in previous sections should assist others, however, who may wish to consider electrodeposition for particular applications.

### 4.1 Effect of Temperature

In addition to those results which we have reported in detail, it was found that thicker coatings of good gloss are deposited at higher temperatures, at least in the range  $10^{\circ} - 30^{\circ}\text{C}$ ; in some commercial installations, in fact, it is recommended that baths be run at  $30^{\circ}\text{C}$  both for this reason and for the added reason that, in production, baths do generate local heating, due to the passage of current through the medium.

## 4.2 Effect of Substrates

Not all surfaces can be satisfactorily coated by anodic electrodeposition with the Baycrl White and Cymel White formulations used. Satisfactory coatings can be deposited on steel, aluminium and tin-plated surfaces by anodic electrodeposition; however anodic electrodeposition of white finishes on copper and on zinc did present difficulties. For copper, during electrolysis, cupric ions are preferentially discharged at the anode and they react with the resin to produce relatively thin, greenish coloured coatings with poor hiding power. With zinc, zinc soaps form and are deposited as thin films with poor adhesion and hiding power. Hence, anodic electrodeposition of copper and of zinc surfaces with Baycrl and Cymel formulations cannot be recommended. On the other hand, cathodic electrodeposition obviates these problems and may present an alternative approach to the deposition of white coatings onto these surfaces.

Phosphated surfaces (21) can also be coated by electrodeposition using Baycrl White and Cymel White formulations. Although the surface is completely covered, the coatings deposited are thinner than those from unphosphated surfaces and they have a rough cast finish. Figs. 11, 12 and 13 indicate that this surface roughness reflects the surface roughness of the uncoated phosphate surface. The lesser roughness in the Cymel White coated surface arises from the ability of the resin to flow during the deposition and stoving cycles, that is, the thermoplasticity of the coating.

## 4.3 Throwing Power : Coating of Steel Cylinders

No discussion of electrocoating and its potential applications can pass over the concept of "throwing power". "Throwing power" of an anionic resin is defined as the ability of the electrodeposition coating to deposit over an entire surface, including recessed areas remote from the cathode. While no specific investigation of the "throwing power" was made, it was noted that coverage of confined surfaces is less than that of surfaces proximate to the cathode or where the electrodeposition paint can circulate freely. This is in accord with previously reported work (22).

As mentioned in the introduction, this investigation was undertaken to ascertain the feasibility of electrodeposition for the coating of small steel cylinders, sealed at one end. Good coverage was achieved over the external surface when the cathode was located outside the cavity of the cylinder but the thickness of the paint film deposited along any thin machining grooves on the inner surface was inadequate. On the other hand, much improved coverage of the internal surface was achieved by locating a second cathode within the cylinder cavity. Some results of these experiments are presented in Table 17.

The use of a second cathode within the cylinder cavity is cumbersome on a production line, however. Hence, further work will have to be carried out to improve the throwing power of the formulated electrodeposition paints so that the recesses of the cylinder are more thickly coated. Some industrial installations overseas operate at applied voltages up to 200 volt, DC; here, relatively thick films are deposited



on the anodic substrate in a very short time, after which the film thickness increases only slowly. Perhaps, in the present situation, the coating of the recessed areas may be improved by carrying out the initial deposition at 60 volts, as is done at present, and then increasing the applied voltage to accelerate electrophoretic migration of the paint resin to these more poorly coated recesses.

#### 4.4 Alternative Materials

As with any process which is being developed for possible production, formulation and reformulation to keep abreast of the supplies and substitute materials becomes necessary.

Although the initial work on Cymel White and Cymel Clear was carried out with Cyanamid Resin XC-4010, this resin preparation has now been reformulated by Cyanamid and is marketed as XC-4012. The original material, Resin XC-4010, was marketed as an acrylic copolymer, 73-76% solids in 2-ethoxyethanol, acid number 95-115; the new material is a very similar resin, supplied as 73-77% solids in butanol, acid number 100-120. One series of experiments with Resin XC-4012 has been carried out and results confirm that the electrodeposition characteristics of this resin, formulated as Cymel White, are very similar to those from Resin XC-4010.

Early experience with formulating Baycrl-based paints led to the conclusion that alkanolamines are preferred to simple amines as the neutralising (or solubilising) base (23). From this work and on subsequent advice from Bayer AG (Germany), the electrodeposition paints were formulated using NN-dimethylethanolamine for this purpose; however this material is not readily available locally and is expensive. On the other hand, local paint manufacturers formulate water borne coatings using a proprietary line, AMP-95 (2-amino, 2-methyl 1-propanol, containing 5% water) (24). It was found that formulations based on Baycrl 36/6 using this amine give a very acceptable white coating of good gloss and film thickness on mild steel, (Fig. 15).

#### 4.5 Production (25,26)

If the process is accepted for production purposes, bath composition should be monitored regularly, supplementary replenishment of resin, pigment and additives will be needed and the conductivity of the bath regulated. Finally, when the bath is spent, due consideration to the disposal of waste material (25) will probably be required. In this latter regard, either the process of ultrafiltration or polymer flocculation may need to be adopted (26).

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## APPENDIX A

### BAYCRYL WHITE 36

#### INGREDIENTS

Baycryn L461W (55 per cent in isobutanol)	160 g
NN-dimethylethanolamine (base)	4.7 g
Rutile titanium dioxide (Tioxide RCR6)	36.0 g
Distilled water	250 ml
Distilled water, satd. with isodecanol	750 ml

#### PREPARATION

The resin, base and pigment were milled overnight with 250 ml distilled water in a porcelain mill, using 6 mm diameter porcelain balls as the grinding and dispersing media. The paint was then thinned with 750 ml distilled water saturated with isodecanol. The pH of the mixture was adjusted to pH 8.1 - 8.2 by the addition of further amine as required.

The solids content is about 10.5 per cent and the pigment : binder ratio of the paint is 0.41.

*Baycryn Clear* was prepared in a similar manner except that no pigment was used.

*Baycryn Red* was formulated using the pigment *Monolite Fast Red 2 GS* (ICI Australia Ltd.). This pigment is an azodye from the coupling of the diazonium salt of 2,4-dinitroaniline with 2-naphthol. The weight of pigment (density  $1.50 \text{ g cm}^{-3}$ ) used was 12.7 g relative to 160 g resin, to give a formulation similar to Baycryn White 36.

## APPENDIX B

### BAYCRYL WHITE 36/6

#### INGREDIENTS

Baycryn L461W (55% in isobutanol)	160 g
NN-dimethylethanolamine (base)	4.7 g
Rutile titanium dioxide (Trioxide RCR6)	36.0 g
Isodecanol	6 ml
Distilled water	250 ml
Distilled water	750 ml

#### PREPARATION

The resin, base and pigment were milled overnight with distilled water, 250 ml, and isodecanol, 6 ml.

The paint was then thinned with distilled water, 750 ml. The pH of the mixture was adjusted to pH 8.1 - 8.2 by the addition of further amine as required.

The solids content of the bath is about 10.3 per cent and the pigment : binder ratio of the paint in the bath is 0.41.

Note: The bath stability of this formulation was superior to that from Baycryn 36, thicker films of higher gloss were deposited but these films had poorer abrasion resistance and poorer adhesion to mild steel. In this sense, the isodecanol appears to be acting as a plasticiser in the deposited film.



## APPENDIX C

### CYMEL ED PAINTS

#### Cymel White

##### INGREDIENTS (2)

Cymel XC-4010 (75% in methylcellosolve)	37.8 g
Cymel XC-1116 Cross-linking Agent	5.8 g
NN-dimethylethanolamine	1.69 g
Rutile titanium dioxide (Tioxide RCR6)	16.5 g
iso-Decanol	3 ml
Distilled water	75 ml
Distilled water	375 ml

##### PREPARATION

The resin, cross-linking agent, base and pigment were milled overnight with distilled water, 75 ml, and isodecanol, 3 ml. The paint was then thinned with distilled water, 375 ml, and the pH of the mixture was adjusted to pH 8.1 - 8.2 by the addition of further amine as required.

The solids content of the bath is about 8 per cent and the pigment : binder ratio of the paint in the bath is about 0.40.

*Cymel Clear* was prepared in a similar manner except that no pigment was incorporated in the formulation.

Note: The bath stability of these Cymel formulations was superior to those from both Baycrl formulations, Baycrl White 36 and Baycrl White 6/36.

#### Dulux Light Grey Electrocoat

The paint as provided was diluted three-fold with distilled water, as directed by the manufacturer's instructions.

TABLE 1  
CONDUCTIVITY MEASUREMENTS

Pigment in Paint			mho cm <sup>-1</sup>
Distilled Water (M.R.L. measurement)			1.75 x 10 <sup>-6</sup>
TiO <sub>2</sub>	RCR6	(Coated, super refined)*	50 x 10 <sup>-6</sup>
	RCR2	(Coated, refined)	51 x 10 <sup>-6</sup>
	RSM2	(Uncoated)	370 x 10 <sup>-6</sup>
	AE	(Anatase)	370 x 10 <sup>-6</sup>

\* All pigments were dispersed in distilled water, at a concentration 10 g pigment per 100 ml water, and allowed to equilibrate at ambient temperature overnight. The pigments were then redispersed immediately before the conductivity measurements were made.



T A B L E 3

CONSTANT VOLTAGE DEPOSITION

RAYCRYL WHITE 36

Deposition Time: 5 minutes

Substrate: Mild Steel Panels

Deposition Voltage (V)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
85	$9.23 \times 10^{-3}$	61 - 66	35 - 37		445
70	$7.48 \times 10^{-3}$	49 - 53	39 - 43		
60	$5.89 \times 10^{-3}$	38 - 41	44 - 46		460
50	$4.79 \times 10^{-3}$	31 - 34	48 - 43		
40	$4.04 \times 10^{-3}$	26 - 28	46 - 49		470

Conditions: (a) Freshly prepared bath, pH 8.1, 20-22°C

(b) Surface area covered:  $147.4 \text{ cm}^2$

TABLE 4CONSTANT VOLTAGE DEPOSITIONBAYCRYL WHITE 30Substrate: Mild Steel PanelsDeposition Voltage: 60 Volt

Deposition Time (min)	Mass deposited per unit area covered (g cm <sup>-2</sup> )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion kg wt x cm <sup>-2</sup>
5	5.60 x 10 <sup>-3</sup>	38 - 41	33 - 39	440	> 2000
3	4.64 x 10 <sup>-3</sup>	27 - 31	42 - 50	470	2000
1	2.94 x 10 <sup>-3</sup>	19 - 20	45 - 54	460	2000

Conditions: (a) Freshly prepared bath, pH 8.1, 19-21°C

(b) Surface area covered: 131.2 cm<sup>2</sup>



T A B L E 2CONSTANT VOLTAGE DEPOSITIONBAYCRYL WHITE 36Deposition Voltage : 60 VoltSubstrate: Mild Steel Panels

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (Z)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5	$6.08 \times 10^{-3}$	40 - 42	40 - 47	2000	460
3	$5.08 \times 10^{-3}$	32 - 34	42 - 49	2000	475
1	$3.15 \times 10^{-3}$	19 - 21	45 - 48	2000	480

Conditions: (a) Freshly-prepared bath, pH 8.1, 21-22°C

(b) Surface area covered:  $136.4 \text{ cm}^2$

T A B L E 3

CONSTANT VOLTAGE DEPOSITION

BAYCRYL WHITE 36

Deposition Time: 5 minutes

Substrate: Mild Steel Panels

Deposition Voltage (V)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
85	$9.23 \times 10^{-3}$	61 - 66	35 - 37		445
70	$7.48 \times 10^{-3}$	49 - 53	39 - 43		
60	$5.89 \times 10^{-3}$	38 - 41	44 - 46		460
50	$4.79 \times 10^{-3}$	31 - 34	48 - 43		
40	$4.04 \times 10^{-3}$	26 - 28	46 - 49		470

Conditions: (a) Freshly prepared bath, pH 8.1, 20-22°C

(b) Surface area covered:  $147.4 \text{ cm}^2$



T A B L E 4CONSTANT VOLTAGE DEPOSITIONRAYCRYL WHITE 30

Substrate: Mild Steel Panels

Deposition Voltage: 60 Volt

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5	$5.60 \times 10^{-3}$	38 - 41	33 - 39	440	> 2000
3	$4.64 \times 10^{-3}$	27 - 31	42 - 50	470	2000
1	$2.94 \times 10^{-3}$	19 - 20	45 - 54	460	2000

Conditions: (a) Freshly prepared bath, pH 8.1, 19-21°C

(b) Surface area covered:  $131.2 \text{ cm}^2$

T A B L E 5

CONSTANT VOLTAGE DEPOSITION

RAYCRYL WHITE 36/6

Deposition Voltage: 60 Volt

Substrate: Mild Steel Panels

Deposition Time (min)	Mass deposited per unit area covered (g cm <sup>-2</sup> )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion kg wt x cm <sup>-2</sup>
5	7.59 x 10 <sup>-3</sup>	45 - 48	59 - 62	1600	410
3	6.10 x 10 <sup>-3</sup>	35 - 38	62 - 65	1600	395
1	4.11 x 10 <sup>-3</sup>	23 - 25	64 - 67	1400	410

Conditions: (a) Freshly prepared bath, pH 8.2, 19-21°C

(b) Surface area covered: 129.3 cm<sup>2</sup>



TABLE 6

## CONSTANT VOLTAGE DEPOSITION

## BAYCRYL CLEAR

Deposition Voltage: 60 Volt

Substrate: Mild Steel Panels

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5	$4.53 \times 10^{-3}$	38 - 40		> 2000	> 340*
3	$4.01 \times 10^{-3}$	30 - 33		> 2000	"
2	$3.31 \times 10^{-3}$	24 - 26		2000	"
1	$2.29 \times 10^{-3}$	18 - 19		2000	"

Conditions: (a) Freshly prepared bath, pH 8.2, 19-20°C

(b) Surface area covered :  $142.8 \text{ cm}^2$ 

(\*) Adhesion measurements were attempted but there was insufficiently strong binding between the adhesive on the dolly of the Twistometer and the deposited film. At  $340 \text{ kg wt x cm}^{-2}$ , the dolly broke away from the surface without bearing the substrate.

T A B L E 7

CONSTANT VOLTAGE DEPOSITION

RAYCRYL RED

Deposition Voltage: 60 Volt

Substrate: Mild Steel Panels

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5		23 - 25	17 - 19	2000	460
3		17 - 19	15 - 17		
1½		14 - 16	20 - 22	1900	470
½		6 - 7	26 - 27	1700	440

Conditions: Freshly prepared bath, pH 8.0 - 8.1, 21-22°C



TABLE 8

CONSTANT VOLTAGE DEPOSITIONBAYCRYL WHITE 36Substrate: Tin Plated PanelsDeposition Voltage: 60 Volt

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5	$6.93 \times 10^{-3}$	48 - 52	38 - 46	1700	
3	$5.54 \times 10^{-3}$	35 - 40	58 - 60		
1	$3.88 \times 10^{-3}$	23 - 27	60 - 63		

Conditions: (a) Freshly prepared bath, pH 8.2, 21-22°C

(b) Surface area covered 112.5  $\text{cm}^2$ Comment: Panels began to buckle at applied torques greater than 150  $\text{kg wt x cm}^{-2}$

T A B L E 9

CONSTANT VOLTAGE DEPOSITION

BAYCRYL WHITE 36

Substrate: Aluminium Panels

Deposition Voltage: 60 Volt

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5	$6.22 \times 10^{-3}$	38 - 43	45 - 48	1600	ca. 300
3	$5.49 \times 10^{-3}$	30 - 34	58 - 62		
1	$3.64 \times 10^{-3}$	18 - 24	59 - 64		

Conditions: (a) Freshly prepared bath, pH 8.2, 21-22°C

(b) Surface area covered: 110.9  $\text{cm}^2$

Comment: Panels began to buckle at applied torques greater than 280  $\text{kg wt x cm}^{-2}$



T A B L E 10

CONSTANT VOLTAGE DEPOSITION

CYMEI WHITE

Substrate: Mild Steel Panels

Deposition Voltage: 60 Volt

Deposition Time (min)	Mass deposited per unit area covered (g cm <sup>-2</sup> )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion kg wt x cm <sup>-2</sup>
5	6.74 x 10 <sup>-3</sup>	40 - 42	66 - 70	1400	420
3	5.97 x 10 <sup>-3</sup>	30 - 34	66 - 68		410
1	3.52 x 10 <sup>-3</sup>	20 - 21	60 - 62	1000	

Conditions: (a) Freshly prepared bath, pH 8.2, 19-20°C

(b) Surface area covered: 105 cm<sup>2</sup>

T A B L E 11

CONSTANT VOLTAGE DEPOSITION

CYNEL CLEAR

Deposition Voltage: 60 Volt

Substrate: Mild Steel Panels

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5		18 - 21		1200	375
3		13 - 15		1100	360
1		8 - 10		900	

Conditions: (a) Freshly prepared bath, pH 8.0, 19°C

(b) Surface area covered: 101  $\text{cm}^2$



T A B L E 12

CONSTANT VOLTAGE DEPOSITIONDULUX LIGHT GREY ELECTROCOATDeposition Voltage: 60 VoltSubstrate: Mild Steel Panels

Deposition Time (min)	Mass deposited per unit area covered ( $\text{g cm}^{-2}$ )	Film thickness (micron)	60° Gloss (%)	Scratch Resistance (g)	Adhesion $\text{kg wt x cm}^{-2}$
5	$1.76 \times 10^{-3}$	7 - 9	17 - 20	425	1800
4	$1.69 \times 10^{-3}$				
3	$1.55 \times 10^{-3}$	7 - 8	18 - 22	400	1800
1	$1.25 \times 10^{-3}$	5 - 7	21 - 26	390	1500
$\frac{1}{2}$	$0.92 \times 10^{-3}$	3 - 5	21 - 23	380	1400

Conditions: (a) Surface area covered:  $78.3 \text{ cm}^2$

T A B L E 13

CONSTANT VOLTAGE DEPOSITION

BATH STABILITY

Deposition Voltage: 60 Volt

Substrate: Mild Steel Panels

Age (days)	Baycrl White 36/6		Cymel White	
	Mass deposited per unit area (g cm <sup>-2</sup> )	Relative Bath Stability	Mass deposited per unit area (g cm <sup>-2</sup> )	Relative Bath Stability
0	7.59 x 10 <sup>-3</sup>	1.00	6.74 x 10 <sup>-3</sup>	1.00
5	5.83 x 10 <sup>-3</sup>	0.77	6.14 x 10 <sup>-3</sup>	0.91
14	5.30 x 10 <sup>-3</sup>	0.70	5.69 x 10 <sup>-3</sup>	0.84
28	4.55 x 10 <sup>-3</sup>	0.60	5.08 x 10 <sup>-3</sup>	0.75
42	4.27 x 10 <sup>-3</sup>	0.56	4.49 x 10 <sup>-3</sup>	0.66

- Note:
1. Surface areas covered: Baycrl 126 cm<sup>2</sup>, Cymel 101 cm<sup>2</sup>
  2. Bath stability has been measured in terms of the mass deposited per unit area (g cm<sup>-2</sup>) as a function of the age of the bath (days).



T A B L E 14

PHYSICAL TESTING OF PAINTS: ADDITIONAL COMPARATIVE DATA

Test	Details (Units)	Electrocoated Paints (Mild Steel Panels)			Spray Coated	
		Dulux	Cymel White	Baycryn 36/6	Epoxy	Acrylic
Scratch	(g)	1800	1400	1600		
Impact (inch lb)	direct	160	160	160	20	60
	reverse	110	160	30	6	60
Adhesion	kg wt cm <sup>-2</sup>	425	420	410	300	70
Bend	Mandrel					
	0.5" (23°C)	p	p	p	s.c.	p
	0.38" (23°C)	p	p	p	f	f
	0.25" (0°C)	p	p	p	f	f
	0.17" (0°C)	s.c.	p	s.c.	f	f
p = pass, s.c. = slight crack, f = fail						
Solvent	Water	p	p	p		
Resistance (24 h)	EtOH	p	p	p		
	Acetone	p,s	f	f		
	CH <sub>2</sub> Cl <sub>2</sub>	p	f	f		
	CuSO <sub>4</sub> (1M)	f,b	p	p		
	HCl (1M)	p	p	p		
	NaOH (½M)	p	p	f,b		
p = pass, s = soften, b = blister, f = fail						

TABLE 15

## SALT SPRAY TEST (200 h)

Panel (a) (Mild Steel)	Assessment of Panel (b)		Assessment of Rust (b)	
	Rust	Blister	Rust	Blister
<u>Baycrl 36</u>				
60/5 (0)	10	10	m	s
60/3 (0)	9	10	h(0.13")	m
60/1 (0)	9	10	h(0.07")	m
60/5 (7)	9	10	m	s
60/1 (7)	8	10	h(0.13")	h
<u>Baycrl 36/6</u>				
60/5 (0)	9	9	m	s
60/5 (7)	9	9	m	s
<u>Cymel White</u>				
60/5 (0)	8	9	h(0.25")	s
60/5 (7)	8	9	h(0.25")	m
<u>Dulux Light Grey Electrocoat</u>				
60/5 (0)	9	10	h(0.13")	h
60/3 (0)	6	3	h(0.13")	m
60/1 (0)	3	3	v.h. (1")	m
60/5 (7)	7	8	v.h. (0.3")	h
<u>Baycrl Clear</u>				
60/5 (0)	9	10	h(0.25")	s
<u>Baycrl Red</u>				
60/5 (0)	8	10	m	v.h.
<u>Reference Paints</u>				
Acrylic	7	10	h	s
Epoxy	9	10	h	s

- Note: (a). Coatings on the individual panels are identified by the coating formulation, the deposition voltage, the time (minutes) of the deposition and the age of the bath (days); 60/5 (7) refers to a coating produced at a voltage of 60 volt applied for five minutes from a bath seven days old.
- (b). The performances are rated on a scale "10-to-0" where "10" indicates no failure, "0" complete failure; s slight, m medium, h heavy and v.h. very heavy rust development.



**TABLE 16**

**ARTIFICIAL WEATHERABILITY OF ELECTRODEPOSITION PAINTS**

Panel (a)	Chalk (b)	Rust (b)	Colour Change (c)		60° Gloss (d)	
			before washing	after washing	Initial (%)	Final (%)
<u>Baycryn 36</u>						
60/5 (0)	9	9	6.8	5.8	33	16
60/3 (0)	9	10	3.6	2.4	42	11
60/1 (0)	9	9	5.0	4.4	46	9
60/5 (7)	9	10	5.9	6.5	35	7
60/1 (7)	9	9	4.1	3.4	48	6
<u>Cymel White</u>						
60/5 (0)	6	10	5.5	5.1	53	8
<u>Dulux Light Grey Electrocoat</u>						
60/5 (0)	10	9	3.6	3.4	21	14
60/3 (0)	8	8	4.1	1.6	18	7
60/1 (0)	8	10	6.3	4.0	14	7
<u>Baycryn Clear</u>						
60/5 (0)	-	9	-	-	-	-
<u>Baycryn Red</u>						
60/3 (0)	8	10	10.1	9.8	13	3
<u>Reference Paints</u>						
Acrylic	10	9	4.4	0.8	64	30
Epoxy	10	9	8.6	5.2	72	27

**Notes:** (a) and (b) refer to the corresponding notes in Table 15.

(c). Colour changes are stated in AN42 units and are calculated from chromaticity measurements, (Ref. 14).

(d). Final gloss measurements were made on the washed panels.

TABLE 17

SURFACE COVERAGE OF MILD STEEL CYLINDERS

Formulation	Baycrl White 36	Cymel White
Cured Deposit (g)	0.90	0.64
Film thickness ( $\mu\text{m}$ )		
outer	55-60	32-38
inner	10-13	6-9

Conditions

Anode : Mild steel cylinder, 35 mm i.d. x 110 mm long, sealed at one end.

External Cathode : Stainless steel panel.

Surface Area of Anode  
    external : 125 cm<sup>2</sup>  
    internal : 95 cm<sup>2</sup>

Deposition Conditions : 60 V applied for 5 minutes.



FIG. 1

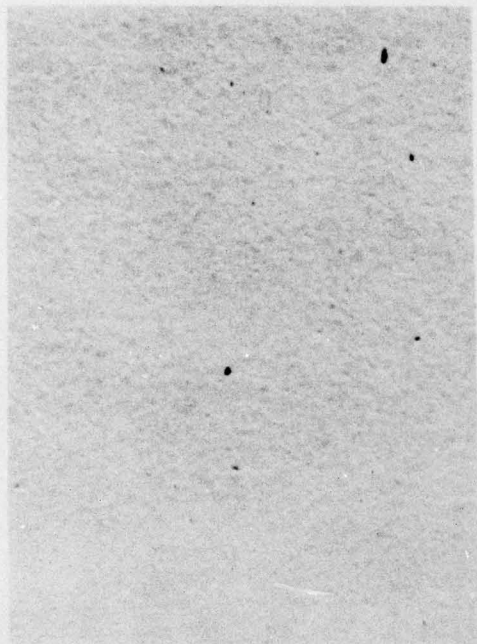
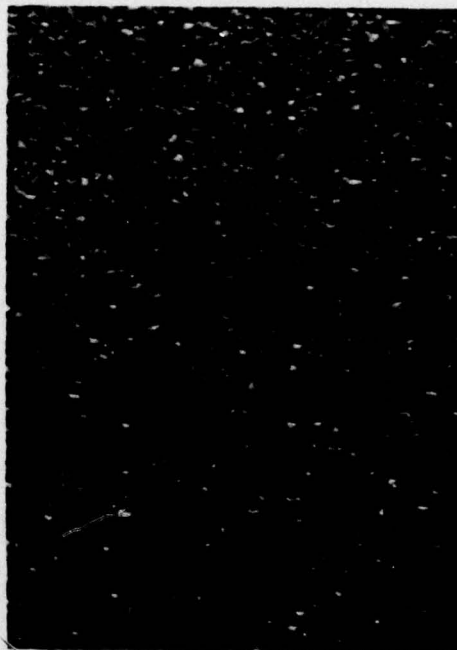


FIG. 2



Micrograph (x25) of the coatings deposited on mild steel panels.

FIG. 1 - Cymel White, deposited at 60 volts applied for five minutes. Film thickness 40-42 micron; Gloss ( $60^{\circ}$ ) 66-70 per cent.

FIG. 2 - Dulux Light Grey Electrocoat, deposited at 60 volts applied for five minutes. Film thickness 7-9 micron; Gloss ( $60^{\circ}$ ) 17-20 per cent.

FIG. 3

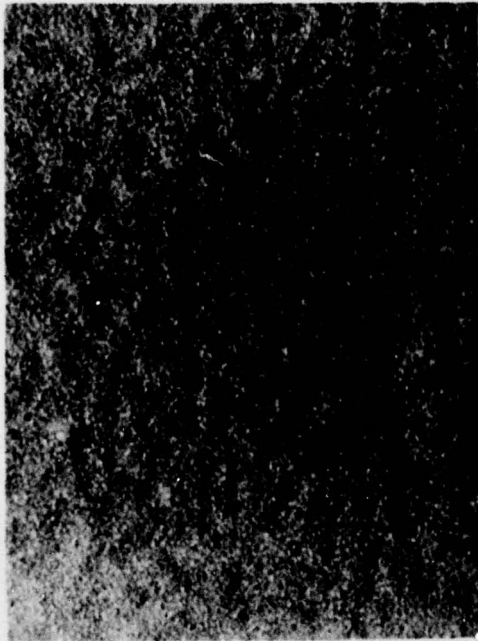
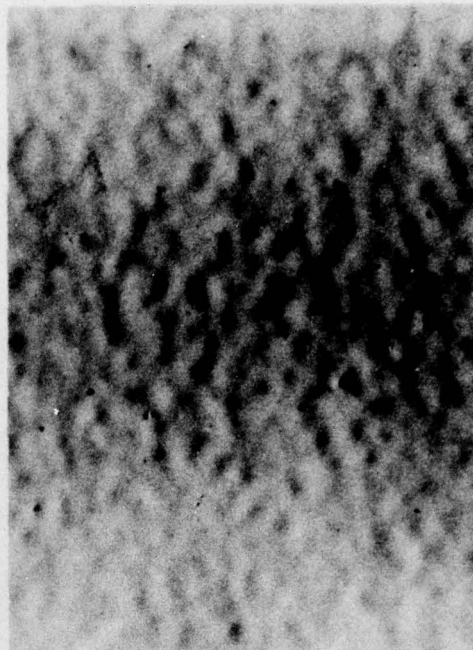


FIG. 4



Micrograph (x25) of the coatings deposited on mild steel panels.

FIG. 3 - Baycryl Red, deposited at 60 volts applied for five minutes. Film thickness 23-25 micron; Gloss ( $60^{\circ}$ ) 17-19 per cent.

FIG. 4 - Baycryl White 36, deposited at 60 volts applied for one minute. Film thickness 19-21 micron; Gloss ( $60^{\circ}$ ) 45-48 per cent.



FIG. 5

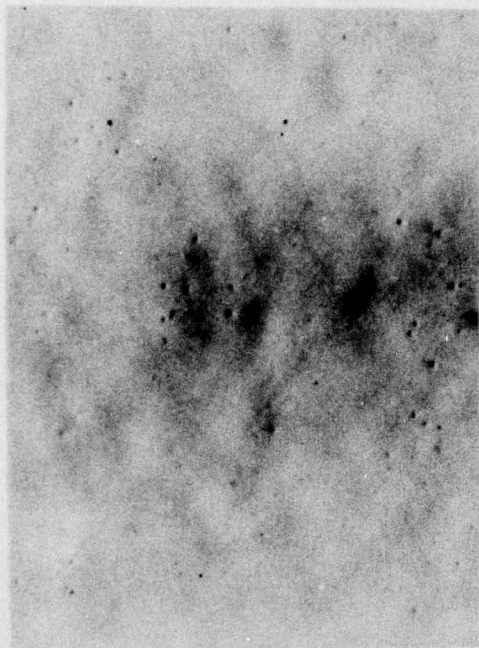
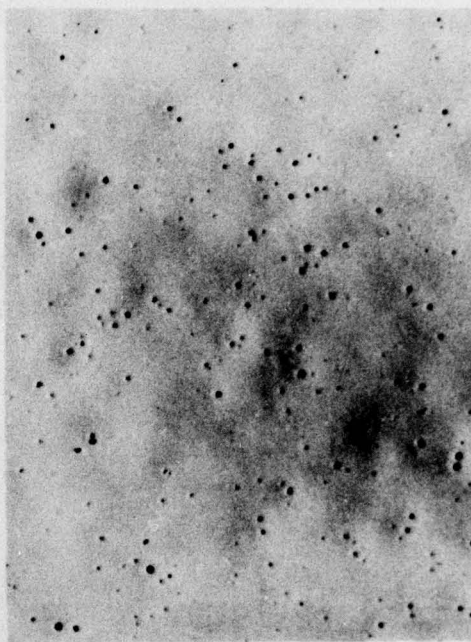


FIG. 6



Micrograph (x25) of the coatings deposited on mild steel panels.

FIG. 5 - Baycryl White 36, deposited at 60 volts applied for three minutes. Film thickness 32-34 micron; Gloss ( $60^{\circ}$ ) 42-49 per cent.

FIG. 6 - Baycryl White 36, deposited at 60 volts applied for five minutes. Film thickness 40-42 micron; Gloss ( $60^{\circ}$ ) 40-47 per cent.

FIG. 7

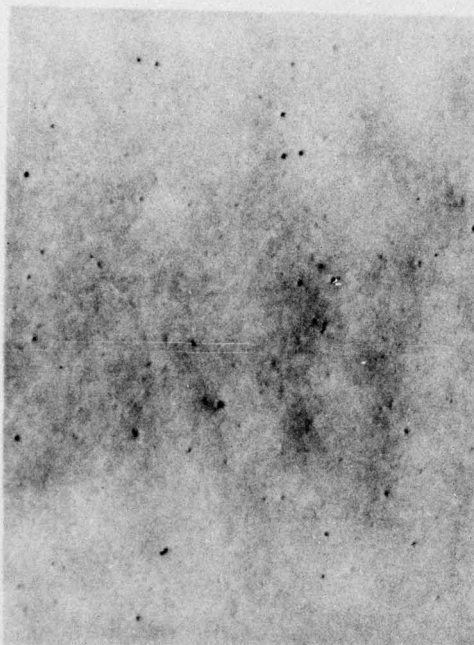
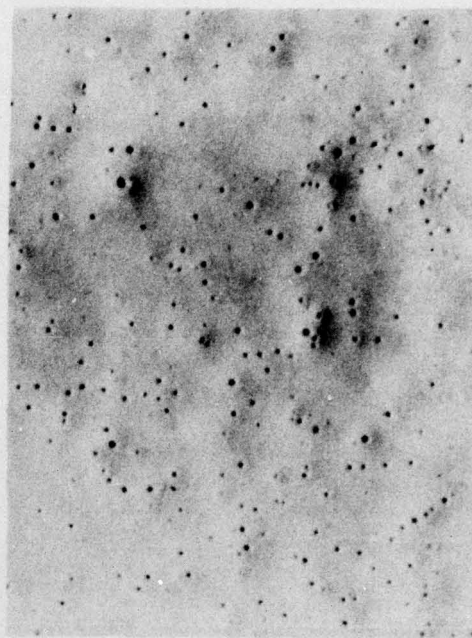


FIG. 8



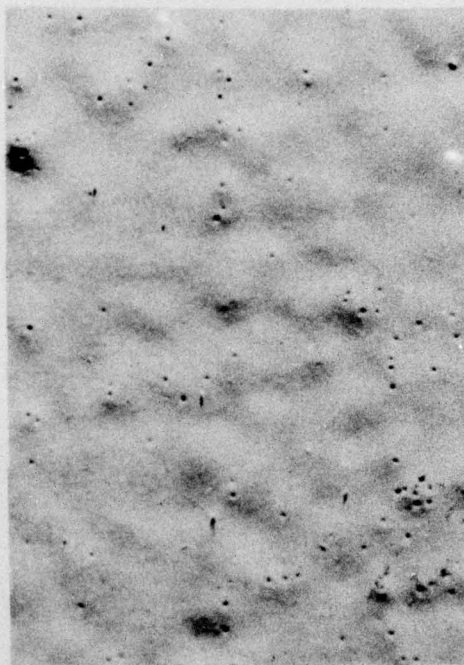
Micrograph (x25) of the coatings electrodeposited on metal surfaces.

FIG. 7 - Baycrl White 36/6 deposited on mild steel with a voltage of 60 V applied for five minutes. Film thickness 45-48 micron; Gloss (60°) 59-62 per cent.

FIG. 8 - Baycrl White 36/6 deposited on tin-plated steel with a voltage of 60 V applied for five minutes. Film thickness 48-52 micron; Gloss (60°) 38-46 per cent.



FIG. 9



Micrograph (x25) of the coatings deposited on metal surfaces.

FIG. 9 - Baycrl White 36/6 deposited on aluminium with a voltage of 60 V applied for five minutes. Film thickness 38-43 micron; Gloss (60°C) 45-48 per cent.

FIG. 10

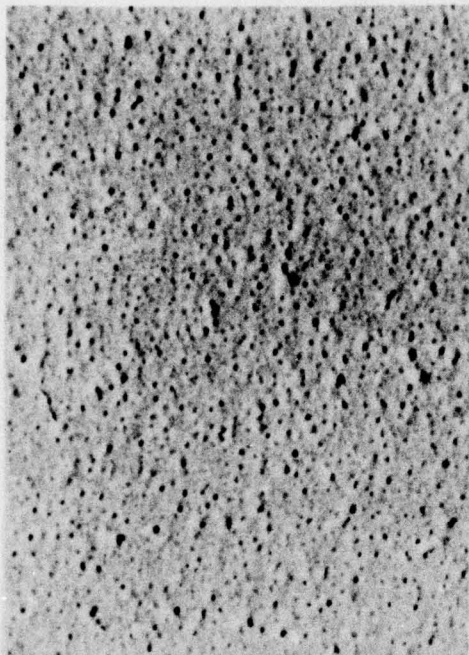
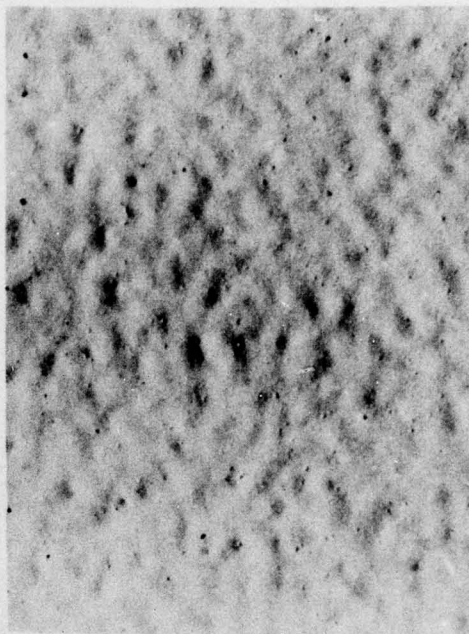


FIG. 11



Micrographs (x25) of the coatings deposited from aged electro-deposition baths onto mild steel panels. Deposition Voltage 60 V applied for five minutes.

FIG. 10 - Baycryn 36, bath age 26 days.

FIG. 11 - Baycryn 36/6, bath age 39 days.



FIG. 12

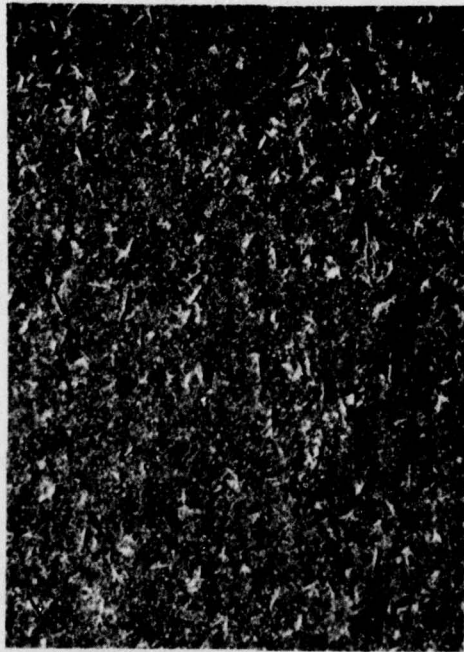


FIG. 12 - Micrograph (x25) of a mild steel panel covered with a phosphate coating.

FIG. 13

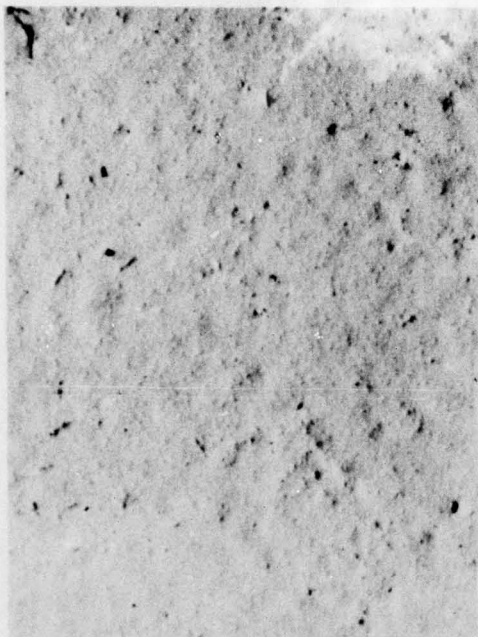
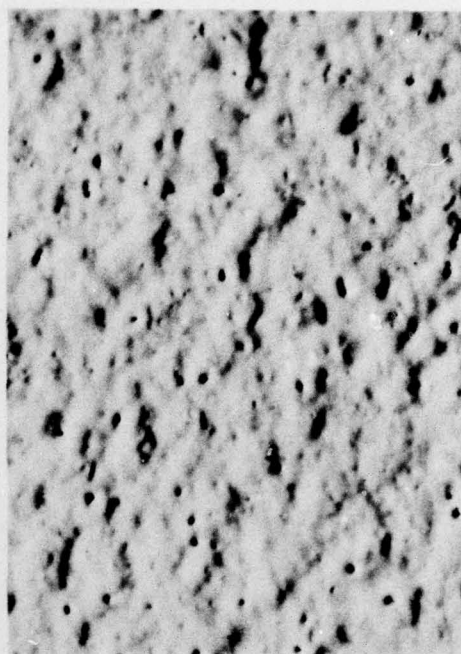


FIG. 14



Micrographs (x25) of the coatings deposited on phosphated mild steel panels.

FIG. 13 - Cymel White, deposited at 60 volts, applied for five minutes.

FIG. 14 - Baycryl 36/6, deposited at 60 volt applied for five minutes.



FIG. 15

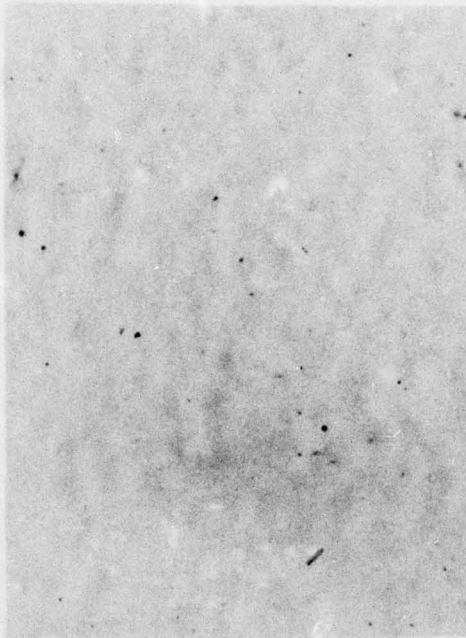


FIG. 15 - Micrograph (x25) of the surface of a Baycrl 36/6 coating using AMP-95 as solubilising base, in the formulation, deposited on a mild steel panel at an electrodeposition voltage of 60 V applied for five minutes.